

MEMORANDUM REPORT BRL-MR-3785

**BRL**

THE MOBILE COMBUSTION DIAGNOSTIC FIXTURE AND ITS  
APPLICATION TO THE STUDY OF PROPELLANT COMBUSTION:

PART I  
INVESTIGATION OF THE LOW PRESSURE COMBUSTION  
OF LOVA XM39 PROPELLANT

J. OMAR DOALI  
ROBERT A. FIFER  
DAVID L. KRUCZYNSKI  
BONITA J. NELSON

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<p>A Mobile Combustion Diagnostic Fixture (MCDF) has been fabricated to investigate the chemical products produced from the combustion of propellants at pressures below 16 MPa. These data are required in order to better understand the combustion phenomenology of propellants at low pressures. The design and capabilities of the MCDF as well as preliminary chemical analysis of nonequilibrium ignition/combustion products using GC and FTIR techniques from hot wire initiated LOVA XM39 propellant are presented.</p> <p>The experiments were performed in both a closed bomb mode and vented chamber mode at various loading densities and pressures. It was found that greater amounts of nonequilibrium combustion products were produced under interrupted burning conditions than under closed bomb conditions and that these products decreased with increasing burst disc pressure and increased with higher loading density.</p>					
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## I. INTRODUCTION

There is presently a need to better understand the phenomenology of the ignition process of propellants, that is the chemical processes occurring at low pressures during propellant combustion. In the past, two-dimensional, two-phase flow interior ballistic models such as TDNOVA<sup>1</sup> have provided improved theoretical gun performance predictions and important charge designer guidelines. However, partly due to a lack of data input for ignition and combustion submodels, these models have assumed a simple surface temperature ignition process followed by a single global energy release at the propellant surface. At this time, newer more sophisticated models such as XKTC<sup>2</sup> require chemical information to more realistically describe igniter-charge interactions. There have been numerous investigations involving the chemical products produced in propellant flames and those produced during propellant decomposition; however, these investigations were usually performed near ambient pressure or under vacuum and did not consider igniter-propellant interactions.<sup>3</sup>

In the past, closed bomb experiments involving weak ignition systems resulted in acoustic oscillations within the chamber. It was thought at the time that the problems were probably due to the gas phase combustion of pyrolysis products.<sup>4</sup> Recently, some experiments using the Ballistic Research Laboratory's 155-mm Howitzer simulator<sup>5</sup> have shown unexpected early stage ignition behavior when a weak igniter and/or low temperature were involved.<sup>5,6</sup> In these experiments acoustic oscillations accompanied a small luminous front observed in the radial ullage between the charge and the chamber wall during early basepad combustion. Subsequently, a very strong luminosity appeared at the forward end of the charge while there was still little visible burning at the base of the charge. In this case, the gas phase combustion of pyrolysis products produced during early stages of combustion was also postulated as the contributing factor. In other experiments involving the interrupted burning of consolidated charges, an exudate was observed on the surface of many unburned propellant grains suggesting a quenched pyrolytic process.<sup>7</sup>

To support these assumptions, it was obvious that an improved understanding of the chemistry occurring in the low pressure region of propellant combustion was necessary. Appropriate analytical techniques such as gas chromatography, infrared spectroscopy, mass spectrometry, and laser spectroscopy were available within the Ballistic Research Laboratory but the question remained as to the approach. Since the above instrumentation was fragile and not designed to be used in the field, a new test fixture was envisioned through the collaboration of various researchers. The final design of the Mobile Combustion Diagnostic Fixture (MCDF) as executed by D. Devynck consisted of a combustion chamber and an expansion chamber separated by a rupture disc.<sup>8</sup> The MCDF was designed to be mobile with a high safety factor (X 10) so that it could be used in various laboratories in close proximity to the analytical instrumentation. The fixture is intended as an instrument to be used in a cooperative research effort involving the Applied Ballistics Branch, the Propulsion Systems Branch, the Ignition and Combustion Branch, and the Advanced Ballistics Concepts Branch of the Ballistic Research Laboratory.

## II. EXPERIMENTAL

### A. Description of the MCDF

The combustion chamber volume is approximately 100 mL depending upon which type of rupture disc is used. The various rupture devices tested will be discussed later. For the purpose of safety, the chamber is designed to withstand a pressure of 350 MPa (50 Kpsi) though the maximum pressure from any experiment will not exceed 35 MPa (5 Kpsi). The chamber is closed at the rear by a threaded plug containing a Kistler 607C2 pressure gage and the firing electrode. Upon rupturing the disc, the gases flow into the expansion chamber which has a volume of 35 liters. This large volume guarantees that once firing occurs, the pressure throughout the fixture will be only slightly above atmospheric pressure. Both the combustion chamber and the expansion chamber may be equipped with either quartz windows or fiber optics to allow application of laser spectroscopic techniques. Additionally, the expansion chamber is equipped with four gas sampling ports. The combustion chamber is also equipped with a relief valve to be used in the event that too small a charge is fired to rupture a disc. A temperature conditioning jacket is also available for the combustion chamber which allows the firing of propellants at other than ambient temperature. The entire fixture is mounted on a trunnion and is completely mobile. A drawing of the fixture is shown in Figure 1. A more detailed description of the MCDF including engineering drawings may be found in Devynck's report. A second mobile unit contains a Nicolet 4094B digital oscilloscope used for data acquisition and other associated equipment including the firing circuits.

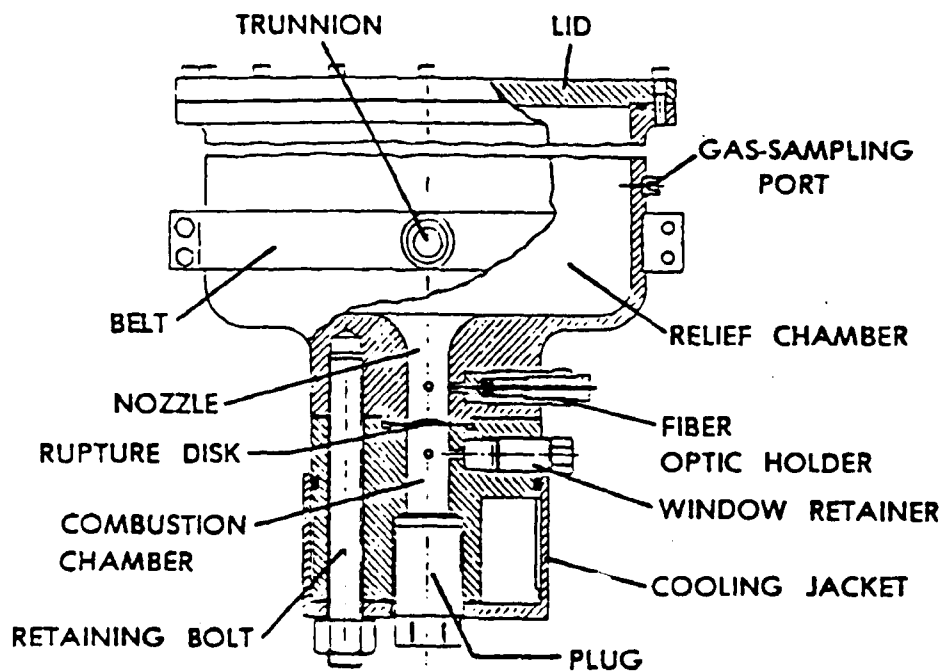


Figure 1. The Mobile Combustion Diagnostic Fixture (MCDF)



## B. Preliminary Tests in the Fixture

Initially, tests were performed by igniting charges of 700X propellant in the chamber using Atlas M100 electric matches with Fike rupture discs rated at 7 MPa (1000 psi). It was found that there was gas leakage where the surface of the expansion chamber met the rupture disc. The fixture was remachined to increase the area of the expansion chamber sealing surface. This apparently solved the leakage problem, however, there were still design problems with the seals associated with the inserts in the vessel's body. A redesign of these seals is in progress. The seal problems did not affect these early experiments but could become critical in the future.

Three different types of rupture discs were tested; those manufactured by Fike Inc., those designed by W. Donovan of the Mechanics and Structures Branch and fabricated in-house, and simple brass shims. The Fike discs were ordered when the chamber was first designed while Donovan's discs were designed because some of the low pressure (3.5 MPa/511 psi) Fike discs did not seal well. These in-house discs were constructed of aluminum with a circular groove cut into one surface and o-rings on each side to insure a good seal. Cross-sectional drawings of the two types of rupture discs are shown in Figure 2. The brass shims were investigated as an inexpensive alternative to the manufactured products. The normal assembling procedure involved cementing the rupture disc onto the combustion chamber using RTV 162, joining the two chambers and torquing the four large bolts to approximately 100 ft-lb. Under these conditions, all of the discs with the exception of the low pressure Fike discs produced good seals. A summary of the results using various rupture discs is given in Table 1.

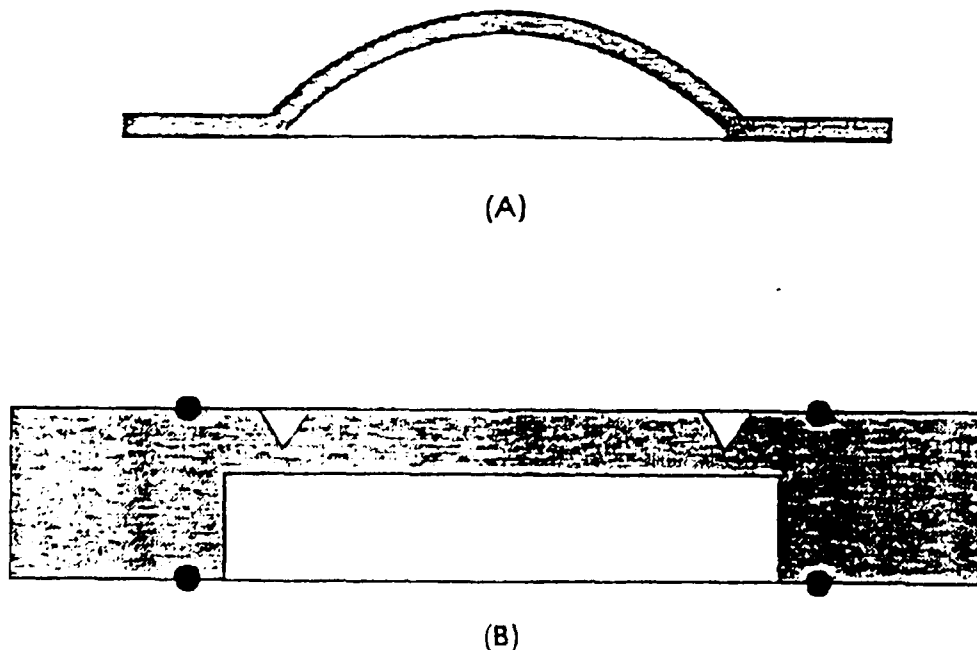


Figure 2 (A)Fike Rupture Disk & (B)Donovan's Rupture Disk

Table 1. Rupture Discs Tests

<u>Rupture Disc (Rating/Thickness)</u>	<u>Observed Pressure MPa(psi)</u>
Fike (6.928 MPa/1005 psi)	6.894(1000)
Fike (6.928 MPa/1005 psi)	7.583(1100)
Fike (17.648 MPa/2560 psi)	18.958(2750)
Donovan (0.355 mm)	10.203(1480)
Donovan (0.355 mm)	9.927(1440)
Donovan (0.241 mm)	7.445(1080)
Donovan (0.241 mm)	7.032(1020)
Fike (35.076 MPa/5088 psi)	42.087(6105)
Brass Shim (0.229 mm)	7.583(1100)

It can be seen that the Fike discs burst near their rated rupture pressures with the exception of the high pressure disc which ruptured approximately 20% higher than expected. The results obtained from the Donovan discs allowed us to estimate dimensions for discs at other burst pressures. Although only one shot was fired using a brass shim, it allowed us to speculate on what thicknesses might be appropriate for future use. All three types of rupture discs were also subjected to tests while in the fixture to determine if their sealing was adequate to maintain a vacuum of at least  $7 \times 10^{-6}$  Mpa (50 mtorr). In all cases, the tests were successful. Volume determinations of the combustion chamber were also made with each type of rupture disc installed so that loading densities could be calculated. The volumes obtained for the Fike, Donovan, and brass shim discs were respectively, 100.0, 99.3 and 89.4 mL.

#### C. Tests Using LOVA XM39 Propellant

In an attempt to reduce propellant vulnerability, LOVA propellants are intentionally difficult to ignite, which may have possible adverse implications for the gun interior ballistic cycle. Reduced flamespreading rates can interact with early projectile motion and alter inbore trajectory, with potential impact on performance and reproducibility. Consequently, the LOVA propellants appear to be important candidates for the study of igniter-propellant interactions using the MCDF. LOVA XM39 propellant (Lot C1-0885-200) was selected for these first investigations since it is pending type classification. A detailed description of the propellant is given in Table 2.

Table 2. LOVA XM39 Description

<u>Composition</u>	<u>Percentage</u>
Cellulose Acetate Butyrate	12.0
Acetyl Triethyl Citrate	7.6
Ethyl Centralite	0.4
Nitrocellulose	4.0
RDX	76.0

<u>Finished Product</u>	
Diameter: 0.724 cm	Density (g/mL): 1.622
Length: 0.858 cm	Total Volatiles (wt %): 0.29
Perf Dia.: 0.038 cm	
Perf No.: 19	
Inner Web: 0.086 cm	
Middle Web: 0.086 cm	
Outer Web: 0.096 cm	

Initial experiments were performed to determine the pressures produced by the combustion of known weights of propellant and the noncondensable products produced under these conditions. For this reason, the fixture was operated as a closed bomb, achieved by installing a rupture disc whose burst pressure (34.525 MPa/5000 psi) exceeded the calculated maximum pressure expected from the sample. In these and other experiments discussed, hot wire ignition was the method of choice in order to determine combustion products without the influence chemical igniters. Chemical igniter and propellant interactions will be investigated in the future. Typical charge configurations involved passing one end of No. 26 Nickel wire through the center perf of each grain while wrapping each grain twice with the other end of the wire. Generally, ignition was achieved by applying a direct current of 18 volts to the wires, however, there were some experiments using rupture discs in which a higher voltage was employed to study the effect of greater energy input. The pressures produced at various loading densities are presented in Table 3.

Table 3. XM39 Firings In Closed Bomb Mode

<u>Grains #</u>	<u>Nominal Weight (g)</u>	<u>Pressure MPa(psi)</u>
1	0.5	0.275(40)
1	0.5	0.827(120)
1	0.5	1.034(150)
2	1.0	3.514(510)
2	1.0	3.348(486)
3	1.5	8.199(1190)
3	1.5	8.819(1280)
4	2.0	13.159(1910)
4	2.0	13.298(1930)

It can be seen that reproducibility at low pressures is poor with this propellant which is not surprising. As a result, the lowest loading density used in future studies involved two grains of XM39. Following these tests at constant volume, a series of experiments were performed using the MCDF equipped with appropriate rupture discs with subsequent chemical analysis. A summary of typical experiments under these conditions is presented in Table 4. A discussion of analytical procedures and results follows.

Table 4. XM39 Firings With Rupture Discs

<u>Grains #</u>	<u>Nominal Weight (g)</u>	<u>Rupture Pressure MPa(psi)</u>
2	1.0	3.219(467)
2	1.0	2.964(430)
4	2.0	3.930(570)
4	2.0	3.840(557)
8	4.0	3.357(487)
8	4.0	3.102(450)
8	4.0	5.136(745)
8	4.0	5.102(740)
4	2.0	8.205(1190)
4	2.0	7.446(1080)
8	4.0	7.860(1140)
8	4.0	6.922(1004)

#### D. Analytical Procedures

During this feasibility study, chemical analysis was limited to measurement of the permanent gases withdrawn from the combustion or expansion chamber. After the propellant and rupture disc were installed, a stainless steel sampling bulb equipped with a syringe septum assembly was connected to the expansion chamber; the expansion chamber and bulb were then evacuated, the valve on the bulb closed, and the expansion chamber filled with UHP Helium (the carrier gas for the GC) to atmospheric pressure. After the firing, the valve was opened to the sampling bulb, which was then installed on a vacuum system equipped with a pressure gage to measure the pressure in the sampling bulb. Helium was then added to the sampling bulb to bring the pressure up to 0.16 Mpa (1200 torr). The procedure was similar for the initial tests in the "closed bomb" mode, except that the pre- evacuated sampling bulb was directly connected to the (air- filled) combustion chamber.

The samples were analyzed by both GC and FTIR techniques. The GC analysis was carried out with a Perkin Elmer Sigma 2000 GC with data station and CHROM II software which automatically produced a report of peak retention times, heights and areas.

One mL injections were made into the GC which contained three columns and two detectors. A Molecular Sieve 5A, 60/80 mesh (3.18mm x 0.93 m ) column and a Porapak Q, 80/100 mesh (3.18 mm x 2.79 m) were connected to a thermal conductivity detector (TCD) while an OV-1701 capillary column (0.25 mm x 30 m) was connected to a flame ionization detector (FID). The two packed columns were connected to the sample and reference sides of the single thermal conductivity detector. The instrument was modified so that the carrier gas from the capillary column injector went through the Porapak column instead of being vented. This permitted the sample from a single injection to be analyzed by both columns simultaneously; a separate injection was made for the molecular sieve column analysis. The GC column oven was programmed as follows: 50 C for one minute, 20 deg/min to 250 C, followed by a 4 minute hold, for a total time of 15 minutes.

This combination of columns and detectors was selected to permit the analysis of a wide variety of ignition/combustion products. The molecular sieve column/TCD combination separates and detects the small permanent gas molecules such as H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>, NO, CO, N<sub>2</sub>O, and CO<sub>2</sub>. The Porapak column does not separate the diatomic molecules, but separates larger volatile molecules including CH<sub>4</sub>, CO<sub>2</sub>, N<sub>2</sub>O, C<sub>2</sub>H<sub>4</sub>, NO<sub>2</sub>, H<sub>2</sub>O, and HCN. The capillary column/FID combination neither separates or detects most of the di- and triatomic molecules, and in general only separates larger, usually condensable molecules, containing 3 or more carbon atoms. The capillary column will be especially useful in future experiments where the MCDF will be directly connected to the GC via a heated transfer line and solenoid valves, permitting analysis of condensable products.

Infrared analysis was also carried out on each sample, using a Digilab FTS-15 Fourier transform infrared (FTIR) spectrometer with a cooled MCT detector, using 400 scans at 2 cm<sup>-1</sup> resolution. After the GC analysis, the gas sampling bulb was connected to an 8 cm pathlength cell equipped with AgCl windows and two filling ports: the valve on the bulb was opened to vent the excess sample pressure through the cell. The FTIR spectrum was then taken. The gases are of course not separated with FTIR as they are with GC, but almost all propellant combustion products can be observed, except for the homonuclear diatomics (e.g., N<sub>2</sub> and H<sub>2</sub>) which are not infrared active. The FTIR analysis was useful in identifying peaks observed with the GC. It is also valuable in the analysis of NO<sub>2</sub>, which is difficult to analyze chromatographically due to decomposition but is easy to measure in the infrared.

### III. RESULTS AND DISCUSSION

The "closed bomb" experiments with sampling employed either 2 or 4 grains, giving maximum pressures of about 2.83 MPa (410 psi) and 8.6 MPa (1250 psi) respectively. These lower pressures were likely due to the dead volume of the connections used to join the sampling bulb to the combustion chamber. The vented chamber tests involved 2, 4 and 8 grain tests at an average rupture disc burst pressure of 3.40MPa (494 psi), and 4 and 8 grain tests at average burst pressures of 5.11 MPa (742 psi) and 7.60 MPa (1100 psi). With the full 35 liter volume of the expansion chamber, dilution of the ignition/combustion products was too great (typically 1000 to 2000 fold). An insert of polyethylene

with an aluminum surface facing the combustion chamber was constructed to reduce the volume of the expansion chamber to about 3 liters for the tests reported here.

Before discussing the observed trends in the measured products, it is useful to inspect the types of chromatograms and FTIR spectra obtained. Figures 3a, 3b, and 3c show the chromatograms (molecular sieve, Porapak and capillary columns) from a vented chamber test with 2 grains and a nominal 3.45 MPa (500 psi) rupture disc with an actual bursting pressure near 3.09 MPa (449 psi). Figures 4a, 4b, and 4c show the chromatograms for a similar experiment using 8 grains of the XM39. Figures 5a, 5b, and 5c show the FTIR spectra for typical 2, 4 and 8 grain experiments at this same burst pressure. The species observed in each chromatogram or spectrum are labeled on the figures. In examining the chromatograms and spectra, the following points should be kept in mind. The response of the thermal conductivity detector (peak height or area) is roughly proportional to the concentration of the molecule; the exception is  $H_2$  which has a thermal conductivity close to that of the Helium carrier gas and therefore gives a very small peak even though present in appreciable concentrations. For the flame ionization detector (FID, capillary column), detector response varies considerably from molecule to molecule, so that calibration is required to quantify the data. This is true also for the FTIR spectra. For example,  $CO_2$ ,  $N_2O$  and  $NO_2$  have very high "extinction coefficients" at their strongest bands, so that relatively small amounts give large bands, whereas  $NO$  and  $CO$  have relatively low extinction coefficients, so that small or moderate band sizes for these products indicate an appreciable concentration.

The chromatograms and spectra show the expected equilibrium combustion products ( $H_2$ ,  $N_2$ ,  $CO$ ,  $CO_2$  and  $H_2O$ ) plus several "nonequilibrium" combustion products ( $HCN$ ,  $CH_4$ ,  $NO$ ,  $N_2O$ ,  $NO_2$  and  $C_2H_4$ ). It is convenient to designate ignition/combustion events as "dirty", "moderately dirty", and "clean" depending on the relative amounts of these "nonequilibrium" products. For example, Figures 3a, 3b, 3c and 5a correspond to "clean" ignition/combustion products, Figure 5b to "moderately dirty", and Figures 4a, 4b, 4c, and 5c to "dirty" products. Any of the four pieces of data (three chromatograms and FTIR spectrum) can be used to assess "dirtiness": For example, the "nonequilibrium" products are quite prominent in the FTIR spectrum (Figure 5c). The capillary column records (Figures 3c and 4c) are interesting in that this column/detector records only "nonequilibrium" products (in this case the H-containing molecules  $CH_4$ ,  $HCN$  and  $C_2H_4$ ) and therefore provides a rapid measure of their amount; note the difference in the height of the (unresolved)  $CH_4/HCN$  peak in the "clean" products (Figure 3c,  $\sim 80$  mV) compared to that for the "dirty" products (Figure 4c,  $\sim 6000$  mV).

The following trends have been observed in the experiments conducted to date with XM39:

a) For the "closed bomb" tests, the products were relatively "clean" (similar to Figures 3a, 3b, 3c and 5a), with only small amounts of "nonequilibrium" products (e.g.,  $HCN$ ,  $CH_4$ ) observed. Somewhat more  $HCN$  and  $CH_4$  was observed for firings with 2 grains than for 4 grains as expected, since the higher pressures from 4 grains should promote more complete reaction under "closed bomb" conditions.

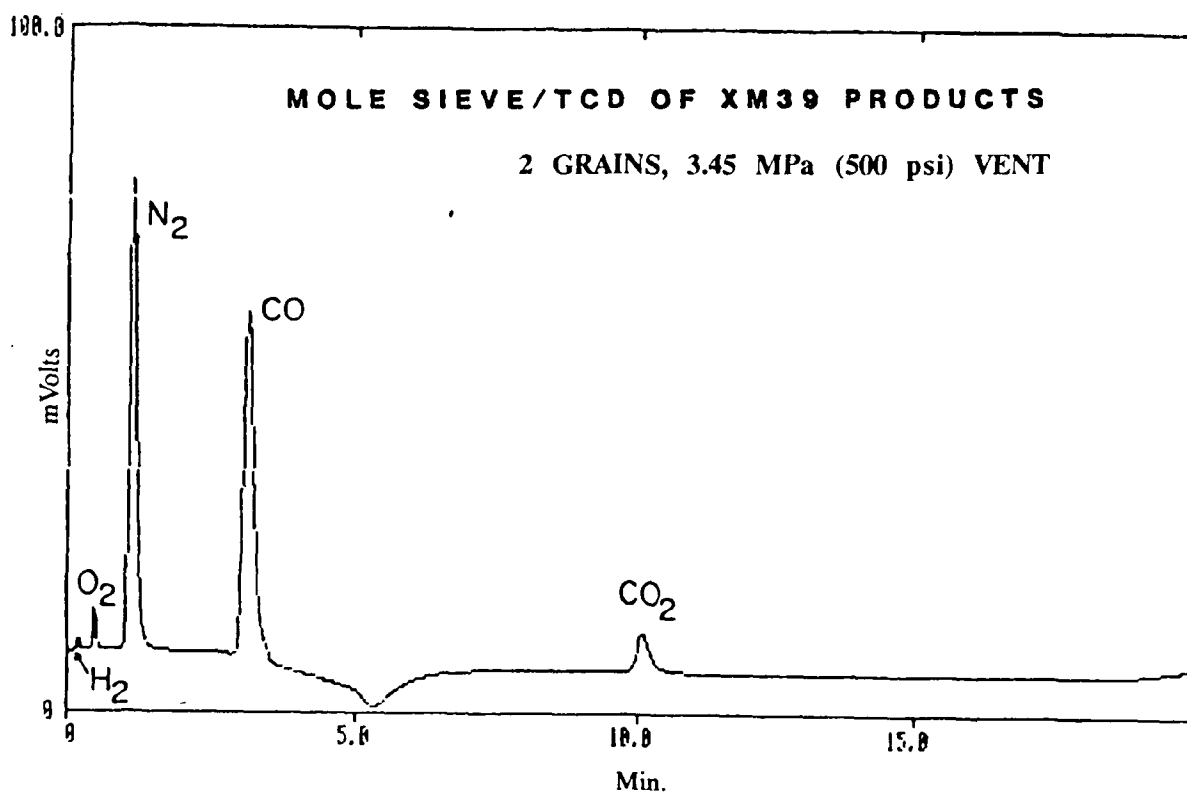


Figure 3a. Gas Chromatographic Separation

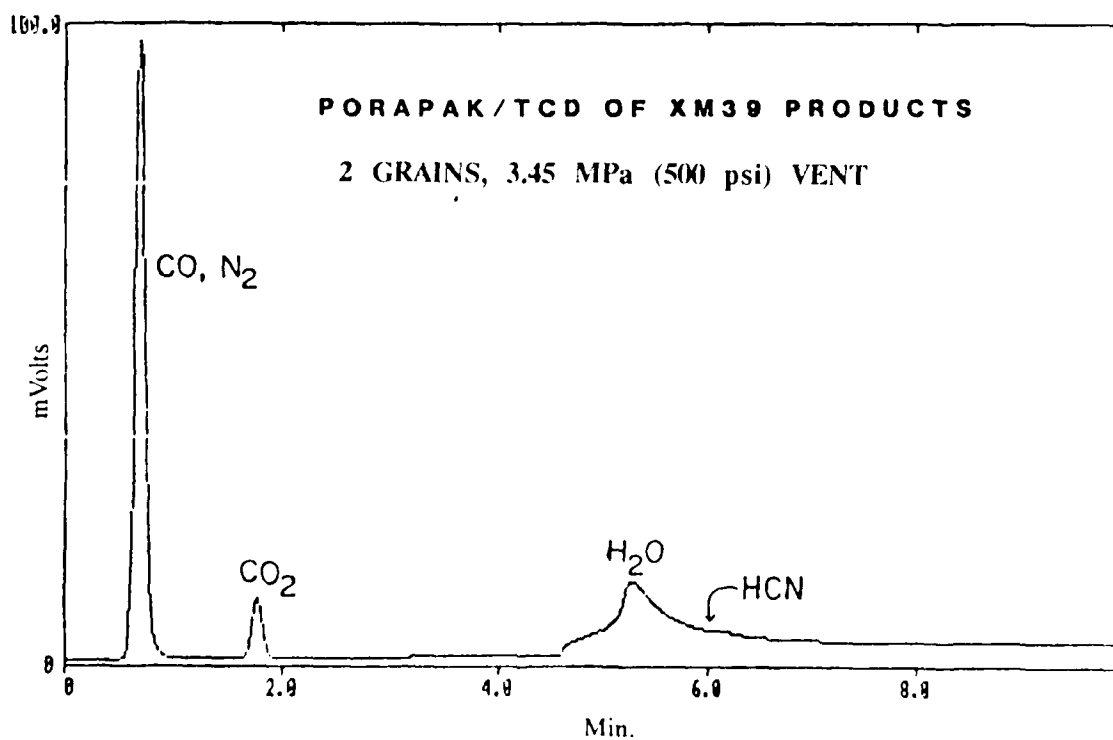


Figure 3b. Gas Chromatographic Separation

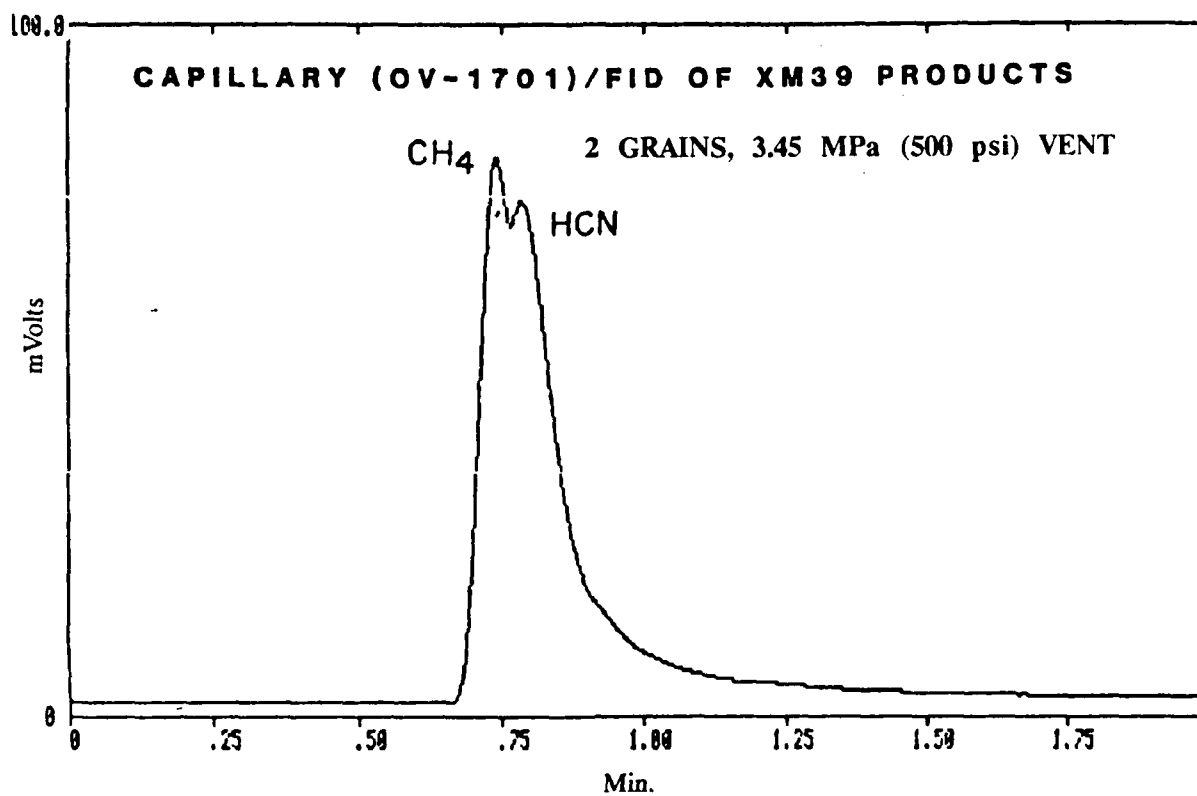


Figure 3c. Gas Chromatographic Separation

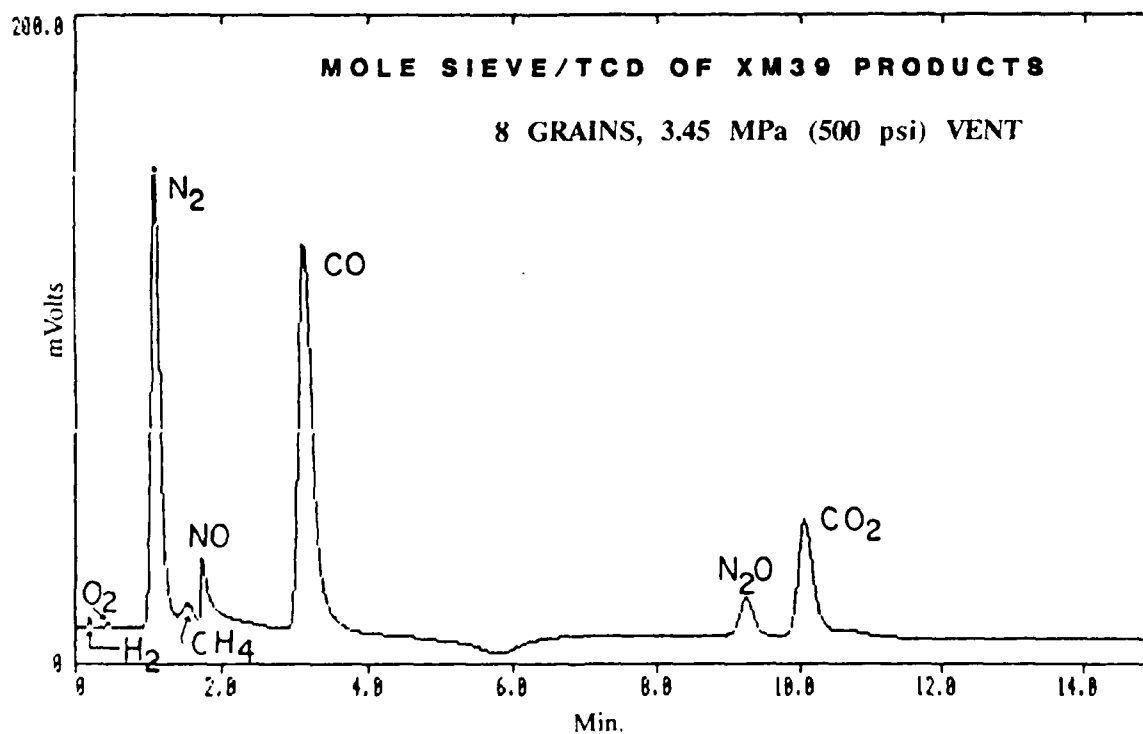


Figure 4a. Gas Chromatographic Separation



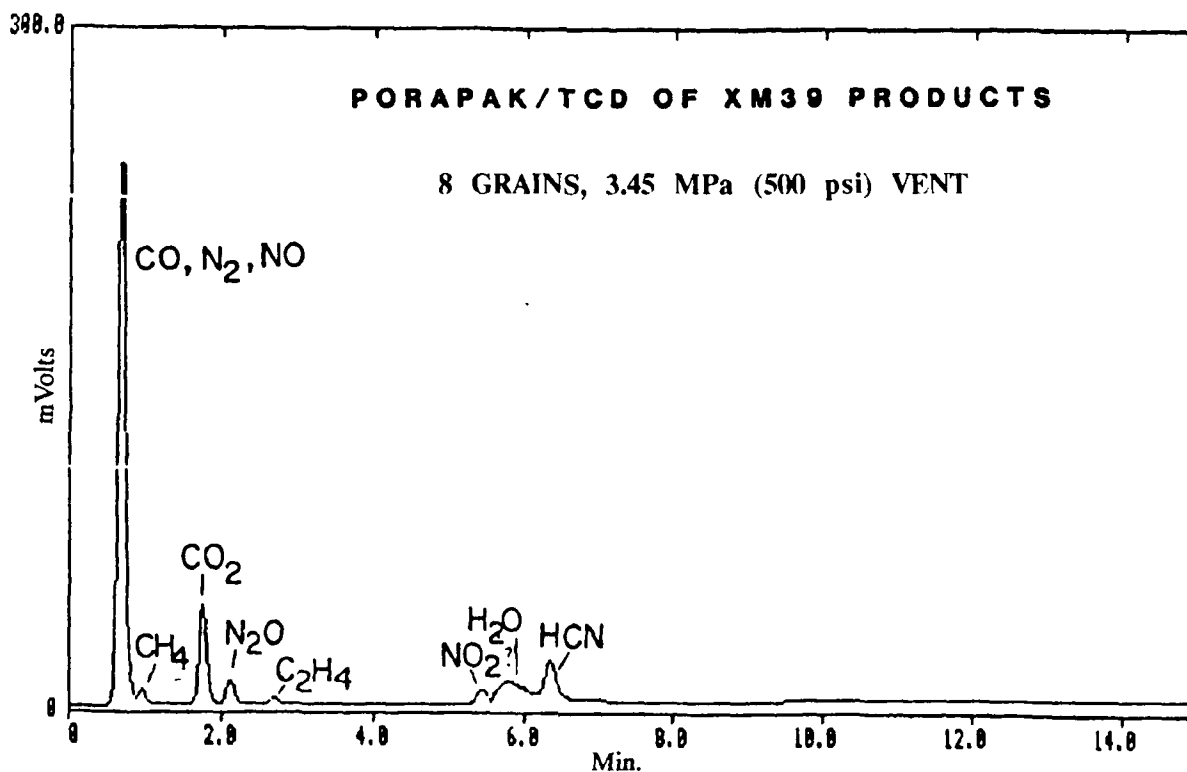


Figure 4b. Gas Chromatographic Separation

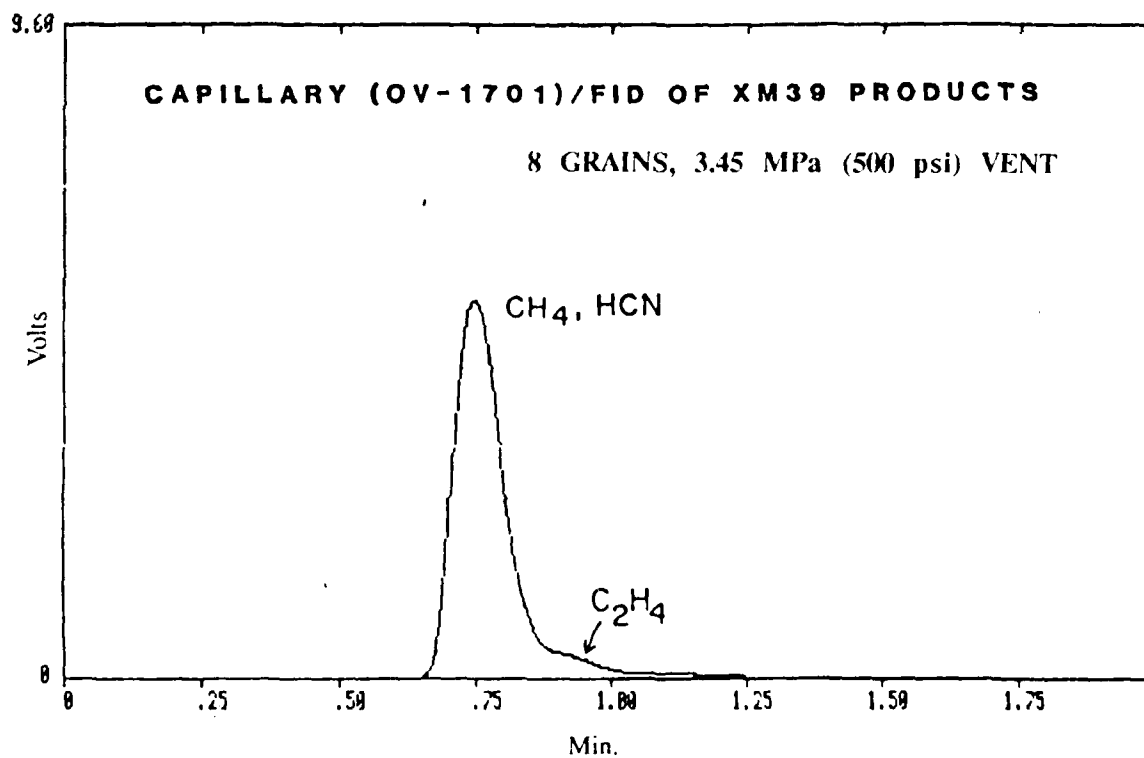


Figure 4c. Gas Chromatographic Separation

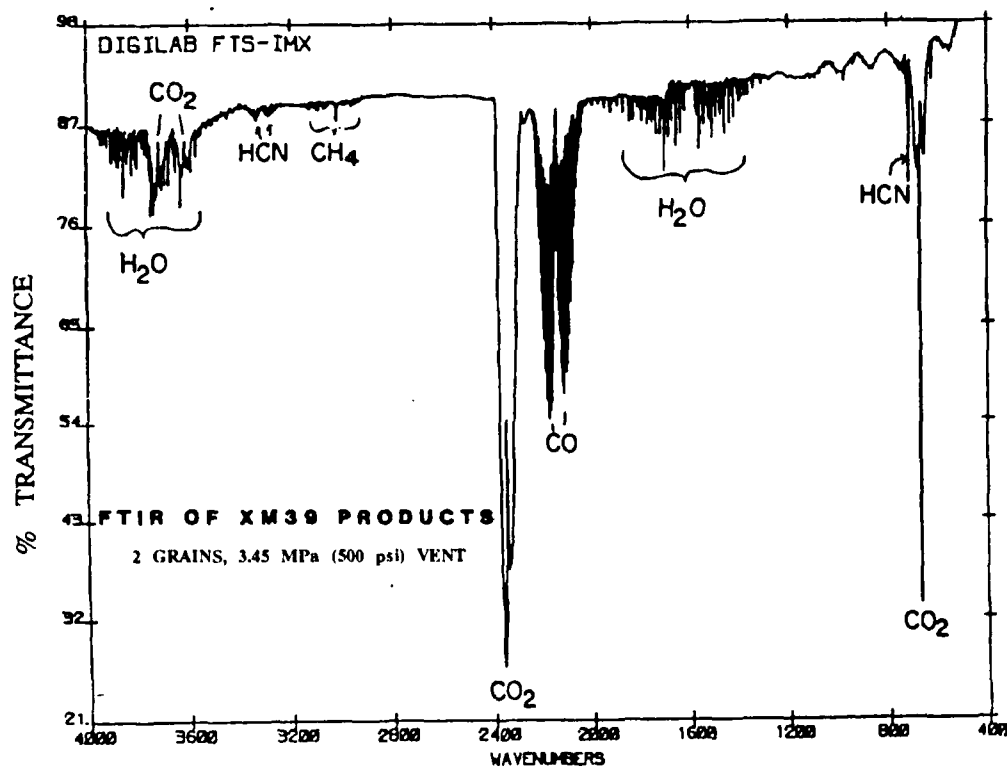


Figure 5a. FTIR Spectra

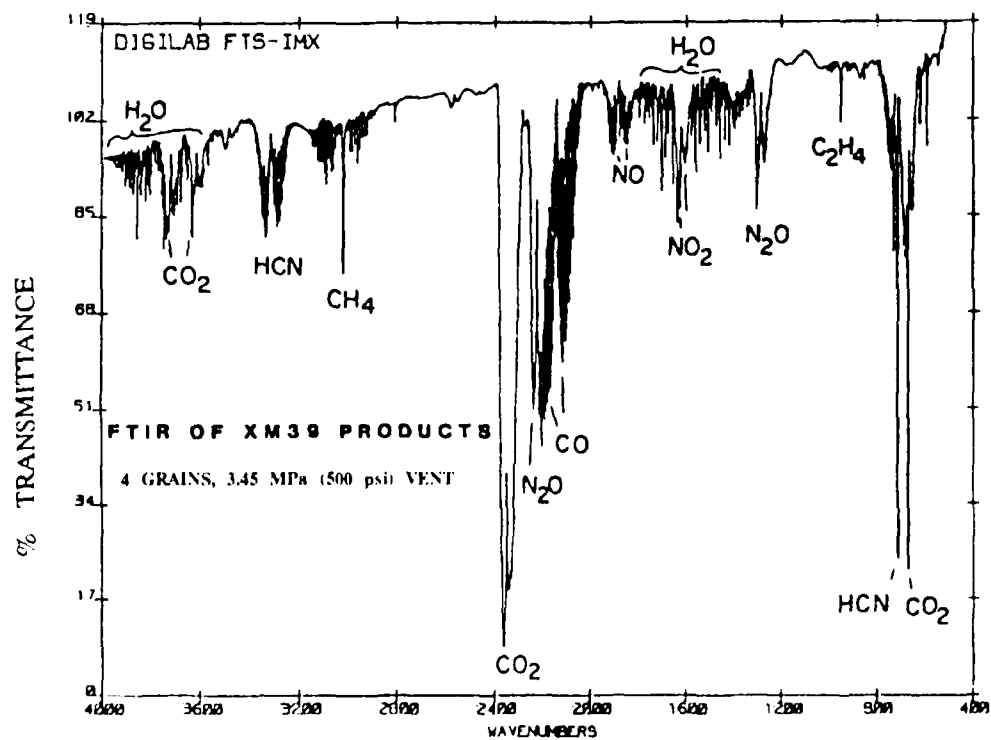


Figure 5b. FTIR Spectra

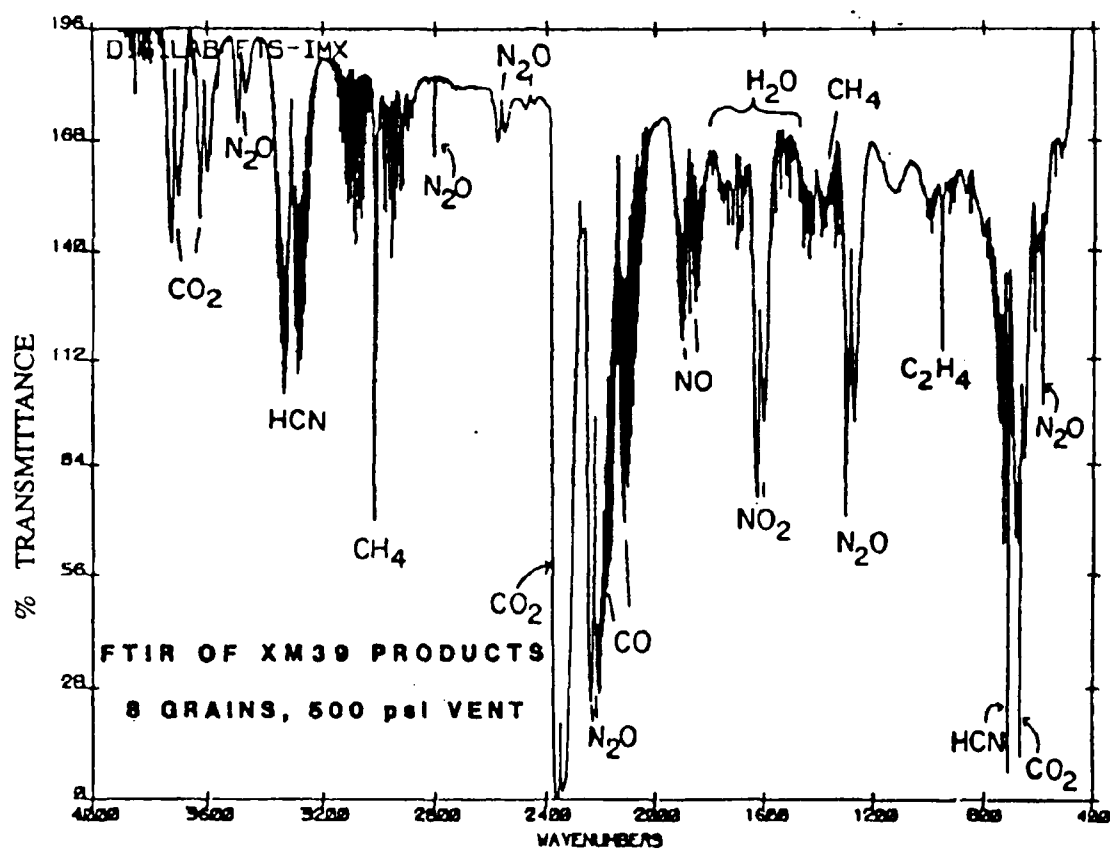


Figure 5c. FTIR Spectra

b) For the "vented chamber" (interrupted burning) test mode, much higher levels of the "nonequilibrium" products were observed than in the closed bomb mode. This confirms the value of vented chamber tests in quenching the "nonequilibrium" products, thereby permitting characterization of delayed/partial reaction chemistry in LOVA propellants.

c) In the "vented chamber" mode, the levels of the "nonequilibrium" products decrease significantly with increase in rupture disc burst pressure, even though the total amount of product gas increases with increasing burst pressure. This is likely a reflection of the longer reaction times and higher pressures available to drive the reactions toward equilibrium. The "nonequilibrium" products were greatest for the nominally 3.45 MPa (500 psi) rupture discs (that actually ruptured closer to 3.23 MPa (470 psi)), and progressively lower for vent pressures of 5.12 MPa (740 psi) and 7.61 MPa (1100 psi). This confirms the importance of the low pressure portion of the ignition process in the delayed energy release phenomenon, as well as the relevance of experiments at a few MPa and less.

d) In the "vented chamber" mode, the levels of the "nonequilibrium" products increase dramatically with increase in the number of grains for a given rupture disc bursting pressure. Figures 3a through 5c illustrate this phenomenon, which was not totally unanticipated, but is not yet fully understood. There are several possible explanations for this phenomenon: a) the higher  $dP/dt$  with the larger number of grains decreases the

available reaction time, b) ignition vs. combustion products: ignition products may be different from combustion products, with a greater proportion of ignition (e.g., pyrolysis) products with a larger number of grains, c) surface effects: the surface of the propellant grains may produce different products from those for the interior of the grains (for a given vent pressure, the depth of burning decreases with increasing number of grains), and d) ignition stimulus effects: with increasing number of grains, the longer ignition wire does not achieve as high a temperature, increasing the amount of pyrolysis products before ignition is achieved. In order to investigate possible effect (c), grains that had been partially burned in one experiment were reignited in a second test; however, the variability of measured products when compared with the original products prevented reaching any conclusions. In order to investigate possible effect (d), a number of 8 grain firings were repeated with higher ignition wire currents to see if the measured products would become "cleaner"; once again, variations of products from run-to-run, did not allow any conclusions. One additional observation that may provide a clue is that the final pressure in the expansion chamber (between 0.10 Mpa [760 torr] and 0.15 Mpa [1140 torr]) is significantly higher for "dirty" ignition than for "clean" ignition. There are, however, two possible explanations for this observation: a) with greater number of grains quenching is not as rapid or complete, and the sample continues to "smolder" after the rupture disc bursts, and b) with more grains, there really is more partial/incomplete chemistry (both the "nonequilibrium" gases measured here and the liquid product that XM39 produces during pyrolysis) prior to venting, and these "nonequilibrium" products generate additional gas during or after the expansion process. Further tests are underway.

e) It is observed that the ratio  $\text{CO}_2/\text{CO}$  (or  $\text{CO}_2/\text{N}_2$ ) increases with increasing amounts of the "nonequilibrium" combustion products for XM39. An increase in this ratio would normally be associated with more complete combustion rather than less complete combustion; the increase in this case may be rationalized as due to the fact that the largest "nonequilibrium" product, HCN, contains no oxygen, thereby increasing the oxygen available for the formation of  $\text{CO}_2$ .

#### IV. CONCLUSIONS

After some modifications, the MCDF functioned as desired. There were no leaks at any of the sealing surfaces at pressures in excess of 41.36 MPa (6000 psi). The Fike rupture discs burst near their rated pressures with the exception of the 34.53 MPa (5008 psi) disc. Rupture discs designed and fabricated in-house also performed well which will eliminate the need to deal with commercial suppliers. For XM39, it was found that the major "nonequilibrium" ignition products are HCN,  $\text{CH}_4$ , NO,  $\text{N}_2\text{O}$ ,  $\text{NO}_2$  and  $\text{C}_2\text{H}_4$ , and that much greater amounts of these partial reaction products are observed under interrupted burning (vented chamber) conditions than under closed bomb conditions, that the amounts of these products decrease with increasing burst disk pressure, and increase with increasing number of grains in the charge. From these preliminary experiments, the MCDF appears to be a promising tool in the investigation of propellant combustion in the low pressure region.

## V. FUTURE PLANS

1. Develop sampling techniques to allow the analysis of any higher molecular weight species.
2. Where applicable, determine the effect of igniters such as Benite, CBI, and the Oxites upon the combustion products under the same conditions.
3. Examine the effect of other than ambient temperatures upon combustion products.
4. Use Laser Spectroscopy to detect any transient species from combustion not observed with more standard methodology.
5. Perform pyrolysis experiments to determine if there is a correlation between these experiments and the results obtained from firings in the MCDF.
6. Investigate the combustion behavior of other selected LOVA, triple-base, double-base, and single-base propellants using the above experimental conditions and procedures.
7. Subject the results from these numerous experiments to cooperative analysis and interpretation with the view to understanding the phenomenology of propellant combustion in the low pressure region and provide data input to ballistic codes so that they more accurately reflect the "real world".

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